

GENERAL COAL REFERENCE MATERIAL ACIRS-G7-2016

CERTIFICATION REPORT

Date of Issue: November, 2016
 Report Number: CR-G7-2016-rev02
 Previous ACIRS-G series: This is the fourth in the series and supersedes ACIRS-G6-2014

Production and certification of this sample was conducted in accordance with ISO 17034 and ISO Guide 35.

ASSIGNED PROPERTY VALUES				
	Property Values¹	Standard Deviation²	Expanded Uncertainty³	Number of laboratories
Ash, % d ^{1a}	9.71	0.09	0.04	37
Volatile Matter, % d ^{1b}	20.32	0.18	0.07	39
Gross Calorific Value, MJ/kg d	32.641	0.073	0.016	137
Relative Density, d ^{1c}	1.371	0.016	0.011	13
Total Carbon, % d	80.19	0.57	0.18	67
Hydrogen, % d	4.45	0.12	0.04	65
Nitrogen, % d	1.82	0.06	0.02	62
Total Sulfur, % d	0.591	0.021	0.004	137
Pyritic Sulfur, % d	0.040	0.016	0.004	21
Sulfate Sulfur, % d	0.01	0.004	0.001	17
Chlorine, % d	0.051	0.004	0.001	49
Phosphorus, % d	0.032	0.002	0.001	14
Fluorine, mg/kg d ^{1d}	96	6	3	22
Mercury, mg/kg d	0.021	0.004	0.002	45
Arsenic, mg/kg d ^{1e}	0.4	0.10	0.07	15
Selenium, mg/kg d ^{1e}	0.6	0.03	0.02	14
Carbonate Carbon, % d ^{1f}	0.06	0.008	0.006	8

	Indicative Values⁴	Standard Deviation²	Expanded Uncertainty³	Number of laboratories
Cadmium, mg/kg d	0.04	0.002	0.001	9

1. Notes

1 Property values are the best estimate of the true value and are based on the robust mean of technically valid results from proficiency test programs using data from the following analysis methods;

- 1a Ash: ISO 1171 and equivalent methods
 - 1b Volatile Matter: ISO 562 and equivalent methods
 - 1c Relative Density: AS1038.21.1.1/1038.21.1.2
 - 1d Fluorine: ISO 11724 and equivalent methods
 - 1e Arsenic and Selenium: ISO 11723 and equivalent methods
 - 1f Carbonate Carbon (Cm): AS 1038.23 and equivalent methods
- All other parameters have been assigned from the results of multiple analysis methods where biases between methods were not observed.

2 Standard deviation (sd) is a robust value used to derive the likely range of results. The value for a measurand from a randomly chosen laboratory would be expected to lay within 2 standard deviations of the certified value with 95% probability.

3 The expanded uncertainty provides the user with information on the likely range of the true (but unknown) value for each parameter and has been estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM) with a coverage factor $k=2$, corresponding to a level of confidence of about 95%. For Cd and Cm, k -factors of 2.3 were chosen according to the t -distribution for sets of results.

4 Indicative values are provided where the relative uncertainty of the robust mean, or distribution of data, was considered unacceptably high and are not considered to be property values.

2. Description of the Sample and Preparation

ACIRS-G7-2016 comprises a sealed jar containing approximately 120 g of coal at a nominal top size of 212 μm . This sample was prepared from 350 kg of a Queensland, Bowen Basin higher rank bituminous coal at -50 mm top size. After a period of stabilisation the coal bulk sample was crushed in a swing hammer mill to a nominal top size of 2.36 mm. The material was then repeatedly mixed by rotary sample division (RSD) until lots of approximately 5 kg were obtained which were then air dried and milled to a nominal top size of 212 μm . This pulverised material was further divided by RSD until representative 120 g samples were obtained. Each sample was then placed into a plastic bag within sealed HDPE jars. Homogeneity of the batch was confirmed by comparison of the dry ash value of each sample against the ash repeatability criteria of ISO 1171.

3. Instructions for Use

This reference material is intended to be used as a quality control tool.

Before the bottle is opened, it **must** be thoroughly mixed by end-over-end rotation so that the material is re-homogenised.

To minimise the risk of compositional changes due to oxidation store in a cool, dark place in original containers with lids tightly sealed. ACIRS cannot be held responsible for any changes that occur after the sample bottle has been opened.

4. Characterisation

ACIRS-G7 was originally tested under Proficiency Testing Australia's (PTA) Round 34 and CANSPEX Q1, 2016 proficiency testing programs. Further testing of ACIRS-G7 was conducted in PTA's Round 36 and ACARP research project C25044 during 2017-18.

The accepted data-set included analyses tested by the following analytical methods;

- Ash: ISO 1171 and equivalent methods
- Volatile Matter: ISO 562 and equivalent methods
- Relative Density: AS1038.21.1.1/1038.21.1.2
- Fluorine: ISO 11724 and equivalent methods
- Arsenic and Selenium: ISO 11723 and equivalent methods
- Carbonate Carbon (Cm): AS 1038.23 and equivalent methods
- All other parameters have been assigned from the results of multiple analysis methods where biases between methods were not observed.

Results were evaluated for technical competency before inclusion in the certification dataset. Generally, data was excluded when:

- Reported from in-house/unknown methods and not within the range of results reported by standard national and international test methods of analysis;
- Results failed to meet technical acceptability along ACIRS guidelines e.g. failing to meet standard method precision limits or identified as outliers from the pooled dataset;
- Significant biases between methods were observed where results from multiple analysis methods were combined.

Robust statistical techniques were then used in the characterisation process in accordance with the guidelines of:

- IUPAC, 2006 International Harmonized Protocol for the Proficiency Testing of Analytical Chemical Laboratories
- ISO 13528-2005, Statistical design for use in proficiency testing by interlaboratory comparison, and
- ISO Guide 35 -2006, Reference Materials – General and statistical principles for certification.

NOTE: The further testing of ACIRS-G7 conducted during 2017-18 resulted in changes to assigned values from the original documentation i.e. inclusion of certified values for Arsenic and Carbonate Carbon; update of the indicative value for Selenium to a certified value; amendment of the certified value for Fluorine and the addition of an indicative value for Cadmium.

5. Period of Validity

Property values for coal samples are subject to change due to the normal oxidation processes for coals. For this reason, oxidation sensitive parameters i.e. calorific value, volatile matter, carbon, hydrogen and forms of sulfur are considered stable until at least November 2019. All other parameters are considered stable until November 2021.

The stability of this sample will be monitored by ACIRS and it is the responsibility of the user to obtain the most recent documentation for this reference material available at www.acirs.com.au/products/general-coal-reference-material

6. Health and Safety

Samples shall be handled in accordance with the Safety Data Sheet available from www.acirs.com.au/products/general-coal-reference-material/

7. Legal Notice

To the extent permitted by law, ACIRS disclaims all warranties whether expressed or implied with regard to merchantability, non-infringement, or fitness for a particular purpose. In no event will ACIRS be liable for incidental damage or consequential loss arising from the use of this product.

Where the product does not conform to assigned property values, giving due consideration to the stated uncertainties and accepted tolerances, the total liability of ACIRS shall be limited at ACIRS' absolute discretion to either replacement of the product or refund of the purchase price.

8. Revision History

Document Number	Summary	Date
CR-G7-2016-rev0	Original (rev0)	14/11/2016
CR-G7-2016-rev1	Updated period of validity	27/03/2017
CR-G7-2016-rev2	Certification update (As, Cd, Cm, F, Se)	14/05/2018

9. Authorisation

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